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(19) AUSTRALIAN PATENT OFFICE

(54) Title
NON-TOXIC, NON-CORROSIVE MICROBICIDAL COMPOSITION

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(57) Claim

1. A non-toxic, non-corrosive, microbicide composition comprising:

(a) a water soluble, short chain organic acid component having the formula:



where R is a short chain carbon compound with a carbon chain length in the range of C₁-C₄, said short chain organic acid component providing an acid pH value in a range of about 1.5 - 4.0 upon dilution with an excess of water;

(b) an intermediate chain length fatty acid component having the formula:



where R' is an aliphatic, straight or branched chain carbon compound with a carbon chain length in the range of C₆--C₁₆;

(c) a non-toxic phenolic compound; and

(d) an effective amount of a solubilizer which is capable of solubilizing the intermediate chain length fatty acid component and the phenolic compound when said microbicide is in a diluted form with water.

AUSTRALIA

Patents Act 1990

PATENT REQUEST: STANDARD PATENT

(Convention Application)

We, being the person(s) identified below as the Applicant, request the grant of a patent to the person identified below as the Nominated Person, for an invention described in the accompanying standard complete specification.

Full application details follow.

[71] Applicant: H.B. FULLER COMPANY
a Corporation duly organised under the laws of the State of Minnesota, United States of America

Address: 2400 Energy Park Drive
St Paul, Minnesota 55108
United States of America

[70] Nominated Person:
Address:

[54] Invention Title: "Non-Toxic, Non-Corrosive Microbicidal Composition"

[72] Name of actual inventor: V Gerold Luss

[74] Address for service in Australia: WRAY & ASSOCIATES, Primary Industry House, 239 Adelaide Terrace, Perth, Western Australia, 6000.

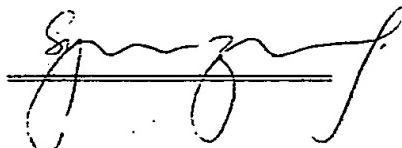
Attorney code: WR

BASIC CONVENTION APPLICATION(S) DETAILS

[31] Application Number	[33] Country	Country Code	[32] Date of Application
08/241,177	USA	US	10th May 1994

Drawing number recommended to accompany the abstract:

H.B. FULLER COMPANY
By its Patent Attorney



5th April 1995
(Date)

P00-008a-b

AUSTRALIA

Patents Act 1990

NOTICE OF ENTITLEMENT

We, **H.B. FULLER COMPANY**, a corporation organised and existing under the laws of the State of Minnesota, United States of America,

of 2400 Energy Park Drive, St Paul, Minnesota 55108, United States of America

being the applicant in respect of Application No 16317/95 state the following:-

NOMINATED PERSON(S)

We are the Nominated Person to whom we request that the patent be granted.

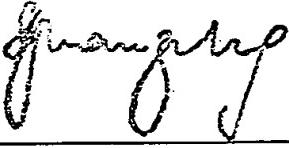
ENTITLEMENT TO INVENTION

The actual inventor is V. Gerold Luss, of 2117-25th Avenue South, Minneapolis, Minnesota, United States of America, and we have entitlement from the actual inventor by virtue of the following facts:

We are the assignee of the inventor in respect of the invention.

CONVENTION APPLICATION

We have entitlement from the applicant of the basic application listed on the patent request form by assignment.


S G KROUZECKY
Patent Attorney
Wray & Associates

29th May 1995

Date

AUSTRALIA

Patents Act 1990

COMPLETE SPECIFICATION

For a Standard Patent

ORIGINAL

TO BE COMPLETED BY APPLICANT

Name of Applicant: H.B. Fuller Company

Actual Inventor: V Gerold Luss

Address for Service:

WRAY & ASSOCIATES, Primary Industry House, 239 Adelaide Terrace, Perth, Western Australia, 6000.

Attorney code: WR

Invention Title: "Non-Toxic, Non-Corrosive Microbicidal Composition"

Details of Associated Provisional Applications Nos:

The following statement is a full description of this invention, including the best method of performing it known to me:-

NON TOXIC, NON CORROSIVE MICROBICIDAL COMPOSITION

Background of the Invention

Field of the Invention

5 This invention relates generally to microbicidal compositions and more particularly to sanitizing and microbicidal compositions which may be used to sanitize surfaces and equipment used to prepare, process or manufacture food, pharmaceuticals or other preparations which require protection from contact with microbial contamination during manufacture.

10

Summary of the Invention

This present invention provides unique non-toxic, non-corrosive sanitizing and microbicidal compositions which may be used to sanitize surfaces and equipment used to prepare, process or manufacture food, pharmaceuticals or other preparations which should receive protection from contact with microbial contamination during manufacture. More generally these compositions may be used wherever there is a desire to substantially reduce or eliminate residual microbial contamination which may reside on equipment surfaces or in ancillary equipment such as valves, pipes, filters, etc. Today the safety of such compositions to humans and/or animals that may come into contact with the composition, its use dilutions or with residuals from the composition which are introduced into the finished product is under increasing scrutiny. The environmental acceptability of such compositions in terms of non-toxicity to life in both treatment plants and ultimate receiving waters is also an important consideration.

25

In brief, the non-toxic, non-corrosive microbicidal compositions of the present invention comprise:

- (a) a water soluble, short chain organic acid component having the formula:



30

where R is a short chain carbon compound with a carbon chain length in the range of C₁--C₄, said short chain organic acid component providing an acid pH value in a range of about 1.5 - 4.0 upon dilution with an excess of water;

- (b) an intermediate chain length fatty acid component having the formula:



where R' is an aliphatic, straight or branched chain carbon compound with a carbon chain length in the range of C₆–C₁₆;

- 5 (c) a non-toxic phenolic compound;
(d) an effective amount of solubilizer which is capable of solubilizing the intermediate chain length fatty acid component (b) and the phenolic compound (c) when the microbicidal composition is diluted with water; and
(e) optional components which may be added to control or enhance foam, aid in
10 control of water hardness, viscosity and stability, or further enhance the antimicrobial activity of the composition.

The short chain organic acid of the composition may be chosen from among the naturally occurring food grade acids generally described as water soluble such as lactic, acetic, citric, malic, succinic, natural amino acids, and the like. In general such acids have
15 four or fewer carbon atoms in their carbon backbone and they may also contain other substituent groups such as –OH, –NH₂, etc., which commonly may be found in such acids. Many such acids have a long history of use in human food and are regarded as either food acidulants or as vital food ingredients (amino acids).

The intermediate chain length fatty acid component of the composition, with a carbon chain length in the range C₆–C₁₆, may include functional groups such as –OH, –NH₂, etc., or may alternatively contain carbon atoms involved in bonds other than sigma bonds. Examples of such acids are caprylic, pelargonic, undecylenic, sorbic, etc., the acids being limited to those which are understood to have a long history of use in food or food related products are non-toxic and recognised as compounds having no adverse effects on
25 humans.

Preferably the phenolic compound of the composition is chosen from that group of compounds which heretofore have been classified as antioxidants and whose advantageous anti-microbial properties have previously been ignored. From this group the preferred subset is the group of anti-oxidants having a long history of use in food products and
30 which belong in the group of compounds "generally recognised as safe" (GRAS).

Microbicidal properties of phenolic compounds have been described in Chapter 9 of *Disinfection, Sterilization, and Preservation*, 3d Ed., Edited by Carl A. Lawrence and Seymour S. Block, Lea & Febiger, Philadelphia, PA, (1983). The use of such compounds in an antimicrobial composition to enhance the antimicrobial activity of said composition
5 is herein described for the first time.

Preferably the solubilizer of the composition is a hydrotrope or a surface active agent chosen from the classes of anionic, cationic, amphoteric or non-ionic surface active agents. The preferred surface active agents are those with a history of use in food and pharmaceutical applications whose toxicity is well understood.

10 As already indicated, a variety of optional components may be added to control or enhance foam, or to aid in controlling water hardness, viscosity and stability, or further enhance the antimicrobial activity of the composition.

Description of the Preferred Embodiments

15 While the invention will be described in terms of its preferred embodiments, it should be understood that this is not intended to limit the invention to those embodiments. On the contrary, it is intended to cover all alternatives, modifications and equivalents which fall within the spirit and scope of the invention as detailed in the appended claims.

The composition is acidic in nature and the major portion of the acidity is provided
20 by the non-toxic short chain water soluble organic acids which have a history of use in food or food related products, are non-toxic and are recognised as compounds which have no adverse effects on humans. In addition, the majority of these acids in their dilute state are non-corrosive to equipment surfaces at the intended use dilutions which are described further hereinbelow for this invention. They are known to be less corrosive than the strong
25 mineral acids which have previously been used in microbicidal solutions. The overall structure of these acids may be represented as:



30 where R is a short chain carbon compound possibly carrying other substituent groups of general carbon chain length 1-4. Examples of such acids are formic, acetic, propionic, butyric, etc. However, the inclusion of substituent groups in the

definition leads to the inclusion of substituted and branched chain acids such as for example:

Lactic Acid R = CH₃CHOH--

Glycolic Acid R = CH₂OH--

5 Alanine R = NH₂CH₂CH₂--

The definition is not confined to the presence of one substituent group since the R group in the original definition may carry multiple substituent groups and even additional acidic groups such as:

Cysteine R = HS--CH₂CNH₂H₂--

10 Malonic, Succinic, Glutaric Acids R = HOOC--(CH₂)_n--

Malonic (n=1), Succinic (n=2), Glutaric (n=3) Acids

The intervening group may again carry substituents such as, for example, in malic or citric acid:

Malic Acid R = HOOC--CH₂CHOH--

15 Citric Acid R = HOOC--CH₂C(OH)(COOH)CH₂--

The second major constituent of the invention is the intermediate chain length organic acid generally described by chain lengths in the range C₆ to C₁₆ which may or may not carry substituent groups. These substituent groups may or may not be additional acid (carboxyl) groups. Examples of such acids are the
20 straight chain fatty acids of general formula,



where R is a straight or branched chain aliphatic group. Examples of such acids are as follows:

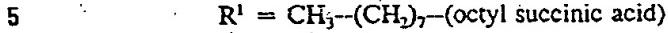
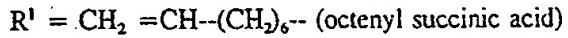
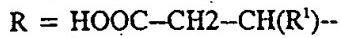
25 R = CH₃--(CH₂)_n--
 Caprylic (n=7) or Pelargonic (n=8)

R = CH₃--C(CH₃)₂--CH₂ CH(CH₃)--
 iso-octanoic acid

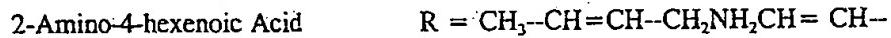
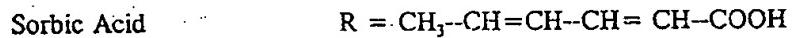
Additionally, the R group may contain 1 or more double or triple bonds which leads to acids of the form such as:

30 R = CH₂ =CH--(CH₂)_n--
 undecylenic acid n=(8)
 decylenic acid n=(7)

The R group may also contain attached substituent groups such as are shown by octenyl succinic acid or octyl succinic acid:



The R group may also contain double or triple bonds in addition to other substituent groups such as for example:

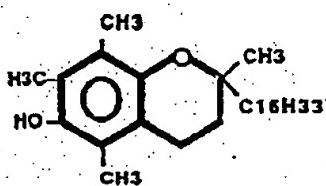


10 Although the above definitions may serve to illustrate the definition of intermediate chain acid they are not meant to be limiting or exclusive of other intermediate chain acids which fall under the general category of acids which are common in foods and are generally considered to be non-toxic.

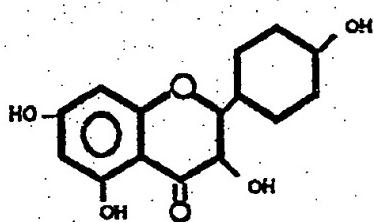
The third and most novel component of the compositions according to
15 the present invention comes from the group of non-toxic phenolic compounds having anti-oxidant properties. Examples of such compounds are BHT (Butylated hydroxy toluene), BHA (Butylated hydroxy anisole), TBHQ (tertiary butyl hydroxy quinone) and natural analogues with similar anti-oxidant properties such as the tocopherols, cinnamic acid compounds and compounds generally described as flavins or

flavonoids. A few such formulae are shown below:

Cinnamic Acid



alpha-Tocopherol



Flavone

5 The use of these phenolic compounds to specifically enhance and increase the efficacy of the sanitizing composition is herein described for the first time.

10 The fourth component of the compositions according to the present invention is a solubilizer. This component may be a surfactant or a hydrotrope which is able to bring and retain all of the components of the composition into solution or stable emulsion, not only in the concentrate but also in the diluted use solutions which are made from the concentrate. The surfactant may be chosen from among the wide range of surfactants that are available including anionic, non-ionic, amphoteric and cationic.

15 The surfactant may be a sulfosuccinate. As is well known to one skilled in the art these compounds based on succinic acid are anionic sulfonated surfactants whose toxicological and environmental properties are well understood. Examples of such compounds are dioctyl sodium sulfosuccinate, commercially available as Solusol[®]FG from American Cyanamid Co., Linden, NJ, dioctyl,

dihexyl, di-isobutyl and ditridecyl sulfosuccinates commercially available under the mark Monawet® from Mona Industries, Inc., Patterson, NJ; diethyl, di-isobutyl, dioctyl and dihexyl sodium sulfosuccinates commercially available under the mark Aerosci® from American Cyanamid Co, Linden NJ; paraffinic and alkylbenzene sulfonate, commercially available under the mark Witconate® from Witco Chemical Company, Houston, TX.

In addition, diphenylether sulfonate adducts commercially available as Dowfax®2A1 from Dow Chemical, Midland, MI, may be used as surfactants.

Not all surfactants may be equally desirable for use in the

compositions of the present invention. From among the anionic surfactants sulfonates in general are preferred over sulfates, as sulfates tend to hydrolyze in acid solutions which may decrease their efficacy as solubilizing agents in liquid concentrates. However, sulfates may be used in compositions which are dry powders, where their stability need only exist over the short period of time during which the prepared use dilution is used.

In addition, the solubilizer component may comprise a combination of surfactants, since a combination of said surfactants may be utilized to optimize useful properties such as rinsability, solubilization, detergency and other properties that may be desirable for the composition to possess.

Optional components may be added to control or enhance foam, or to aid in controlling water hardness, viscosity and stability. In cases where it may be desirable, one may also add solvents such as limonene, to enhance the cleaning action of the composition and also enhance penetration into biofilms, soils, and the like. As is well known to those skilled in the art, one may also optionally add encapsulating agents which encapsulate one or more of the components and permit either the formation of an emulsion or the formation of a dry powder. Chelating agents may be added to enhance biological activity, cleaning performance and control water hardness. In those cases where it is desirable one may also add fragrances, dyes and bittering agents to enhance the composition for its intended use. Additional components which further enhance the antimicrobial activity may also be added.

THE COMPOSITION AND EXAMPLES

A general formula for a use dilution of the composition is shown in Table 1.

TABLE 1	
5	Intermediate Chain Length Organic Acid
	40-140 ppm
	Phenolic (Anti-Oxidant, flavone)
	25-75 ppm
	Surfactant/Hydrotropes
	20-120 ppm
10	Water Soluble Organic Acid
	400-1000 ppm
10	Water
	balance

The novelty of the composition described in Table I resides in the unique admixture of components. Each component, intermediate chain length fatty acid, phenolic, surfactant and water soluble organic acid, is known to have some measure of antimicrobial activity. None, by itself, is capable of meeting the requirements for a sanitizing composition at the use dilutions and conditions set forth herein. However, taken in combination, they function synergistically at high dilutions with the desired efficacy.

It is well known in the scientific literature that low pH values generated by acidic solutions provide an environment hostile to the growth of microorganisms.

Previous patents, such as U.S. Patent No. 4,404,040 issued Sept. 13, 1983 to Wang, for example have described the use of short chain organic acid solubilized by anionic surfactants in such an acid environment to form a sanitizing composition. It is known in the art that mineral acids such as phosphoric acid are a preferred acidifying vehicle. The present composition, however, recognizes that water soluble organic acids and phenolic compounds may be used to augment the efficacy of these other components.

The present composition also recognizes that the overall anti-microbial efficacy of the composition is enhanced by the inclusion of these water soluble organic acids and phenolics, thereby permitting a lower concentration of the other components. The inclusion of these water soluble organic acids and phenolics in a

highly acidic composition for killing microorganisms is therefore a novel aspect of the present composition.

The advantageous use of such components in a sanitizing composition, their synergistic interaction with the other components in the relatively acidic environment of these sanitizing compositions, their non-corrosive nature, in addition to other useful properties which enhance the use of acidic sanitizing compositions is herein described for the first time.

Exemplary concentrates of the composition, based on percent composition, were prepared and are shown in Table 2.

10

TABLE 2.

15

20

EXAMPLE	1	2	3	4	5	6	7
Pelargonic Acid	5	10	10	8	8	8	8
TBHQ	3	3	3	3	3	3	--
BHA	--	--	--	--	--	--	3
Lactic Acid	87	50	52.5	45	55	55	50
Citric Acid	--	--	--	10	--	--	--
Solusol FG	5	--	--	--	--	--	--
Monawet MO 70	--	5	--	--	--	--	5
Aerosol A-268	--	--	5	--	--	--	--
Witconate NAS-8	--	--	--	7	6	7	--
Dowfax 2A1	--	--	--	3	3.5	2.5	--
Water	--	32	29.5	24	24.5	24.5	34
TOTAL	100	100	100	100	100	100	100

25

The method used for determining anti-microbial activity was the Germicidal and Detergent Sanitizers Test accepted by the USDA and the EPA. The method was taken from Methods of Analysis, Association of Official Analytical

Chemists, 11th edition (1970) pp 66-68. The use concentrations or dilutions of the above formulas were all at 1 fluid ounce to 5 gallons of water.

The results of these tests conducted in hard water as specified in the test (500 ppm as CaCO₃) on compositions 1-7 of Table 2 are shown in Table 3.

5

TABLE 3

EXAMPLE	1	2	3	4	5	6	7
E. Coli ATCC 11299 kill	pass						
10 Staph. Aureus ATCC 6538 kill	pass						

In this testing an entry of pass indicated that the indicated composition when tested showed a 99.999% or greater kill within 30 seconds of contacting the microorganisms being tested.

15

Previous microbicidal compositions do not contemplate the substantial advantages to be gained from the synergistic functioning of the subject compositions.

20

The composition described above include components which are known to have some measure of antimicrobial activity, although none of these active ingredients is capable of meeting the requirements for a sanitizing composition at the use dilutions and conditions described herein. However, taken in combination, they function synergistically at high dilutions with the desired efficacy.

The herein described compositions also have other desirable properties which include low corrosivity, minimal environmental impact, and primarily contain materials which are often present in foods.

25

This completes the description of the preferred and alternate embodiments of the invention. Those skilled in the art may recognize other equivalents to the specific embodiment described herein which equivalents are intended to be encompassed by the claims attached hereto.

WHAT IS CLAIMED IS:

1. A non-toxic, non-corrosive, microbicide composition comprising:

(a) a water soluble, short chain organic acid component having the formula:



5 where R is a short chain carbon compound with a carbon chain length in the range of C₁–C₄, said short chain organic acid component providing an acid pH value in a range of about 1.5 - 4.0 upon dilution with an excess of water;

(b) an intermediate chain length fatty acid component having the formula:



where R' is an aliphatic, straight or branched chain carbon compound with a carbon chain length in the range of C₆–C₁₆;

15 (c) a non-toxic phenolic compound; and
(d) an effective amount of a solubilizer which is capable of solubilizing the intermediate chain length fatty acid component and the phenolic compound when said microbicide is in a diluted form with water.

2. A composition as claimed in claim 1 wherein the short chain acid component (a) is selected from the group consisting of formic acid, acetic acid, propionic acid, butyric acid, lactic acid, glycolic acid, adipic acid, glycine, glutamic acid, alanine, phenylalanine, cysteine, malonic acid, succinic acid, glutaric acid, malic acid, citric acid, hydroxyacetic acid, and mixtures thereof.
3. A composition as claimed in claim 1 wherein the short chain organic acid is an organic dicarboxylic acid.
- 25 4. A composition as claimed in claim 1 wherein the short chain organic acid is an amino acid or amino acid derivative.
5. A composition as claimed in any one of the preceding claims wherein the short chain acid component is a liquid composition.

6. A composition as claimed in any one of claims 1 to 4 wherein the short chain acid component is a solid composition.
7. A composition as claimed in any one of the preceding claims wherein the intermediate chain length fatty acid component (b) is selected from the group consisting of caprylic acid, pelargonic acid, iso-octanoic acid, decylenic acid, undecylenic acid, decanoic acid, heptanoic acid, hexanoic acid, octenyl succinic acid, isobutyl succinic acid, octyl succinic acid, sorbic acid, 2-amino-4-hexenoic acid, n-octyl succinic acid, n-nonyl succinic acid, caprylic acid, pelargonic acid, undecylenic acid, sorbic acid, octenyl succinic acid, n-octenyl succinic acid, n-10 nonenyl succinic acid, hydroxycinnamic acid, cinnamic acid, sorbic acid, phenyl acetic acid, and 2-Amino-4-hexenoic acid and mixtures thereof.
8. A composition as claimed in any one of the preceding claims wherein the intermediate chain length fatty acid component has a carbon chain length of 7-14.
9. A composition as claimed in claim 8 wherein the intermediate chain length fatty acid has a carbon chain length of 8-10.
10. A composition as claimed in any one of the preceding claims wherein the intermediate chain length acid has an unsaturated carbon bond located in the carbon chain.
11. A composition as claimed in claim 10 wherein the intermediate chain length acid carries multiple unsaturated bonds.
12. A composition as claimed in any one of the preceding claims wherein the non-toxic phenolic compound (c) is selected from the group consisting of BHT (Butylated hydroxytoluene), BHA (Butylated hydroxyanisole), TBHQ (tertiary butylhydroquinone), PG (propyl gallate), alpha-tocopherol, delta-tocopherol, 15 pelargonidin, peonidin, malvidin, anthocyanins, cinnamic acid compounds, flavins, flavinoids and mixtures thereof.
13. A composition as claimed in any one of the preceding claims wherein the phenolic compound is an antioxidant.
14. A composition as claimed in any one of the preceding claims wherein the 20 solubilizer (d) is selected from the group consisting of surfactants, hydrotropes and mixtures thereof.

15. A composition as claimed in any one of the preceding claims wherein the solubilizer is selected from the group consisting of sulfates, sulfonates, sulfosuccinates, phosphate esters, anionic sulfated surfactants and mixtures thereof.
16. A composition as claimed in claim 15 wherein the solubilizer is a sulfonate based on paraffinic hydrophobes or hydrophobes containing benzene rings.
17. A composition as claimed in any one of claims 1 to 14 wherein the solubilizer is a nonionic surfactant selected from the group consisting of ethylene oxide, ethylene oxide/propylene oxide adducts of ethylene glycol, alkyl glycals, parafinic alcohols, substituted phenols and mixtures thereof.
- 10 18. A composition as claimed in claim 17 wherein the substituted phenol is selected from the group consisting of octyl phenol and nonyl phenol.
19. A composition as claimed in any one of claims 1 to 14 wherein the solubilizer is an amphoteric surfactant selected from the group consisting of alkyl imidazolines, alkyl amines and mixtures thereof.
- 15 20. A composition as claimed in any one of the preceding claims further comprising an additive selected from the group consisting of solvents, encapsulating agents, fragrances, dyes, bittering agents and mixtures thereof.
21. A composition as claimed in claim 20 wherein the additive is limonene or a terpene.
- 20 22. A composition as claimed in claim 1 prepared as a dry powder concentrate.
23. A composition as claimed in any one of the preceding claims prepared in dilute form with added water.
24. A composition as claimed in claim 23 diluted with water to a concentration wherein the short chain acid component (a) is in a range of about 45-90%; the intermediate chain length fatty acid component (b) is in a range of about 5-10%; the non-toxic phenolic compound (c) is in a range of about 1-5%; and the solubilizer is in a range of about 2-10%.
25. A composition as claimed in claim 23 diluted with water to a concentration wherein the short chain water soluble organic acid component is present at a range of about 400-1000 ppm, the intermediate chain length organic acid is present at a range of about 40-140 ppm, the phenolic compound is present at a range of about 25-75 ppm, and the solubilizer is present at a range of about 20-120 ppm.

26. A composition as claimed in claim 1 prepared as an emulsion.
27. A non-toxic non-corrosive microbicide composition substantially as herein described in the description of the preferred embodiments and examples.

Dated this Fifth day of April 1995.

H B Fuller Company
Applicant

Wray & Associates
Perth, Western Australia
Patent Attorneys for the Applicant.

NONTOXIC, NONCORROSIVE MICROBICIDAL COMPOSITION

Abstract Of The Disclosure

- 5 The invention provides environmentally acceptable sanitizing and microbicidal compositions that are safe to humans and/or animals, which may be used to sanitize surfaces and equipment used to prepare, process or manufacture food, pharmaceuticals or other preparations which should receive protection from contact with microbial contamination during manufacture. These compositions
- 10 substantially reduce or eliminate residual microbial contamination which may reside on an equipment surface or in ancillary equipment such as valves, pipes, filters, and the like. The present concentrated compositions comprise a water soluble short chain organic acid component with a carbon chain length in the range C₁-C₄ which provides an acid pH value in a range of about 1.5 - 4.0 upon dilution with an excess
- 15 of water, an intermediate chain length fatty acid component with carbon chain length in the range C₆-C₁₆, an anti-oxidant phenolic compound component, a solubilizer component chosen from hydrotropes and surfactants, present in an effective amount for solubilizing the intermediate chain length fatty acid component and the phenolic component when the microbicidal concentrate is in a diluted form with water, and
- 20 optional components which may be added to control or enhance foam, and aid in control of water hardness, viscosity and stability.

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